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IS 7729 (1975): Sodium Monochloroacetate [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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*Indian Standard*  
SPECIFICATION FOR  
SODIUM MONOCHLOROACETATE

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# Indian Standard

## SPECIFICATION FOR SODIUM MONOCHLOROACETATE

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# *Indian Standard*

## SPECIFICATION FOR SODIUM MONOCHLOROACETATE

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 8 July 1975, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

**0.2** Sodium monochloroacetate is used in the manufacture of sodium carboxymethyl cellulose (CMC), carboxymethyl starch (CMS), ethylene diaminetetraacetic acid (EDTA) and carboxymethyl guar gum. It is also used in the manufacture of herbicides like 2,4-Dichlorophenoxyacetic acid.

**0.3** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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### 1. SCOPE

**1.1** This standard prescribes the requirements and methods of sampling and test for sodium monochloroacetate (SMCA).

### 2. REQUIREMENTS

**2.1 Description** — The material shall be white to creamish amorphous or granular powder.

**2.2 Solubility** — The solubility of the material in distilled water at 25°C shall be about 38 percent (38 percent SMCA and 62 percent water).

**2.3** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

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\*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR SODIUM MONOCHLOROACETATE

( Clause 2.3 )

Sl No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST ( REF TO CL No. IN APPENDIX A )
(1)	(2)	(3)	(4)
i)	Sodium monochloroacetate, percent by mass, <i>Min</i> , on dry basis	96.0	A-2
ii)	Sodium chloride (as NaCl), percent by mass, <i>Max</i> , on dry basis	1.5	A-2
iii)	Free monochloroacetic acid, percent by mass, <i>Max</i> , on dry basis	1.0	A-2
iv)	Sodium dichloroacetate, percent by mass, <i>Max</i> , on dry basis	1.0	A-2
v)	Sodium carbonate ( as $\text{Na}_2\text{CO}_3$ ), percent by mass, <i>Max</i> , on dry basis	0.5	A-3
vi)	Sodium glycollate, percent by mass, <i>Max</i> , on dry basis	1.5	A-4
vii)	Moisture, percent by mass, <i>Max</i>	2.0	A-5
viii)	Iron salts ( as Fe ), percent by mass, <i>Max</i>	0.004	A-6

### 3. PRECAUTIONS IN HANDLING

**3.1** Sodium monochloroacetate causes irritation to the skin. Proper care should, therefore, be exercised while handling this material.

### 4. PACKING AND MARKING

**4.1 Packing** — The material shall be packed in polyethylene lined bituminized hessian bags provided with extra polyethylene bags.

**4.2 Marking** — The bags shall be securely closed and shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and/or his recognised trade-mark, if any;
- Lot or batch number; and
- Net mass of the contents.



#### 4.2.1 The bags may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

### 5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to this standard shall be judged as prescribed in Appendix B.

## APPENDIX A

( Clause 2.3 and Table 1 )

### METHODS OF TEST FOR SODIUM MONOCHLOROACETATE

#### A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070 - 1960\* ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2. DETERMINATION OF SODIUM MONOCHLOROACETATE

##### A-2.1 Reagents

A-2.1.1 *Sodium Hydroxide Solution* — 5 N.

A-2.1.2 *Nitric Acid* — 5 N.

A-2.1.3 *Phenolphthalein Indicator Solution* — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-2.1.4 *Standard Silver Nitrate Solution* — 0.1 N.

\*Specification for water, distilled quality ( revised ).

A-2.1.5 *Concentrated Nitric Acid* — conforming to IS : 264 - 1968\*.

A-2.1.6 *Ferric Alum Indicator Solution* — saturated.

A-2.1.7 *Standard Ammonium Thiocyanate Solution* — 0.1 N.

A-2.1.8 *Nitrobenzene*

## A-2.2 Procedure

A-2.2.1 *Determination of Total Chlorine* — Weigh 4 g of the dried material ( see A-5.2.2 ) in a 100-ml glass-stoppered flask and add 30 ml of sodium hydroxide solution. Reflux the contents under a water-cooled condenser for 2 hours, using glass beads to prevent bumping. After cooling, rinse the condenser with a small quantity of distilled water and neutralise the contents with nitric acid, using phenolphthalein as indicator. Transfer the solution to a 100-ml one-mark graduated flask, add distilled water to the mark and shake well. Name the solution as *Solution A*. Transfer 10 ml of the *Solution A* to a conical flask and then add 5 ml of nitric acid in excess. Add 50 ml of standard silver nitrate solution, 3 ml of nitrobenzene and shake vigorously. Titrate the contents with standard ammonium thiocyanate solution using ferric alum as indicator, until a permanent faint reddish-brown colour appears. Carry out a blank titration using the same amount of reagents and following the same procedure.

### A-2.2.1.1 Calculation

$$\text{Total chlorine content ( } X \text{ ), percent by mass} = \frac{35.45 (V_1 - V_2) N}{M}$$

where

$V_1$  = volume in ml of standard ammonium thiocyanate solution required for the blank titration,

$V_2$  = volume in ml of standard ammonium thiocyanate solution required in the titration with the material,

$N$  = normality of standard ammonium thiocyanate solution, and

$M$  = mass in g of the dried material taken for the test.

A-2.2.2 *Determination of Sodium Chloride Content* — Weigh 3 to 4 g of the dried sample ( see A-5.2.2 ) in a 250-ml flask, neutralize with concentrated nitric acid and then add 5 ml of the acid in excess. Add 20 ml of standard silver nitrate solution. Further add 3 ml of nitrobenzene and shake vigorously. Titrate the contents with standard ammonium thiocyanate solution using ferric alum indicator until a permanent faint reddish-brown colour appears.

\*Specification for nitric acid ( *first revision* ).

**A-2.2.2.1 Calculation**

Chlorides (as NaCl), percent by mass ( $X_1$ ) =  $\frac{5.845 (V_3 - V_4) N}{M_1}$

where

$V_3$  = volume in ml of standard ammonium thiocyanate solution required for the blank titration,

$V_4$  = volume in ml of standard ammonium thiocyanate used in the titration with the material,

$N$  = normality of standard ammonium thiocyanate solution, and

$M_1$  = mass in g of the dried material taken for the test.

**A-2.2.3 Determination of Free Acid ( Monochloroacetic Acid )**

**A-2.2.3.1 Reagent** — Standard sodium hydroxide solution — 0.1 N.

**A-2.2.3.2 Procedure** — Weigh about 5 g of the dried material (see A-5.2.2) in a 250-ml conical flask. Dissolve in 50 ml of distilled water and titrate the contents with standard sodium hydroxide solution using phenolphthalein indicator.

**A-2.2.3.3 Calculation**

Monochloroacetic Acid ( $X_2$ ), percent by mass =  $\frac{9.45 \times V_5 \times N_2}{M_2}$

where

$V_5$  = volume in ml of standard sodium hydroxide solution used in the titration,

$N_2$  = normality of standard sodium hydroxide solution, and

$M_2$  = mass in g of the dried material taken for the test.

**A-2.2.4 Determination of Sodium Dichloroacetate****A-2.2.4.1 Reagents**

- Calcium acetate solution* — 5 percent.
- Concentrated ammonium hydroxide solution.*
- Sulphuric acid solution* — 2 N.
- Standard potassium permanganate solution* — 0.1 N.

**A-2.2.4.2 Procedure** — Transfer 50 ml of solution *A* (see A-2.2.1) in a 250-ml beaker and add 10 ml of calcium acetate solution. Heat to boiling and neutralize with concentrated ammonium hydroxide solution adding a few ml in excess. Place the beaker on a water-bath for about 1 hour and allow the contents to stand overnight at room temperature. Filter off the precipitate on filter paper and wash with dilute ammonia. Return the precipitate to the beaker and remove the last traces of the precipitate from the filter paper by washing with 50 ml of sulphuric acid. Heat the contents to 70°C and titrate with standard potassium permanganate solution to a faint rose-colour which persists for 30 seconds.

#### A-2.2.4.3 Calculation

$$\text{Sodium dichloroacetate } (X_3), \text{ percent by mass} = \frac{30.18 \times V_6 \times N_3}{M_3}$$

where

$V_6$  = volume in ml of standard potassium permanganate solution used in the titration,

$N_3$  = normality of standard potassium permanganate solution, and

$M_3$  = mass in g of the material taken for the test.

#### A-2.3 Calculation

Sodium monochloroacetate, percent by

$$\text{mass} = 116.45 \left[ \frac{(V_1 - V_2)N}{M} - \frac{(V_3 - V_4)N}{10M_1} - \frac{V_5 \times N_2}{10M_2} - \frac{V_6 N_3}{2.5M_3} \right]$$

where the legends are the same as given in A-2.2.1.1, A-2.2.2.1, A-2.2.3.3 and A-2.2.4.3.

### A-3. DETERMINATION OF SODIUM CARBONATE

#### A-3.1 Reagents

**A-3.1.1 Ammonium Hydroxide Solution** — Sp gr 0.90.

**A-3.1.2 Barium Chloride Solution** — 10 percent (*m/v*), neutral to phenolphthalein indicator.

**A-3.1.3 Standard Hydrochloric Acid** — 0.1 N.

**A-3.1.4 Standard Sodium Hydroxide Solution** — 0.1 N.

**A-3.1.5 Phenolphthalein Indicator** — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit conforming to IS:323-1959\* and dilute to 100 ml.

\*Specification for rectified spirit (*revised*).

**A-3.2 Procedure** — Weigh 10 g of the dried material (see A-5.2.2) and dissolve in 50 ml of water containing sufficient ammonia to keep the solution slightly alkaline, add another 3 ml of ammonia solution in excess and then add while stirring 15 ml of barium chloride solution. Cover the beaker with a thin rubber membrane to keep away the carbon dioxide in the air. After 30 minutes, filter the solution and wash the precipitate with 50 ml of diluted barium chloride solution and then with 20 ml of water. Transfer the precipitate along with the filter paper to a washed beaker, add 50 ml of water and a few drops of phenolphthalein indicator. Titrate the contents with standard hydrochloric acid until the red colour has disappeared and then add another 30 ml of standard hydrochloric acid to completely dissolve the precipitate. Boil the contents for a few minutes, cool and titrate the excess hydrochloric acid with standard sodium hydroxide solution.

### A-3.4 Calculation

$$\text{Sodium carbonate (as Na}_2\text{CO}_3\text{), percent by mass} = \frac{5.3 (V_1 N_1 - V_2 N_2)}{M}$$

where

$V_1$  = volume in ml of standard hydrochloric acid used in the titration,

$N_1$  = normality of standard hydrochloric acid,

$V_2$  = volume of standard sodium hydroxide solution used in the back titration,

$N_2$  = normality of standard sodium hydroxide solution, and

$M$  = mass in g of the dried material taken for the test.

## A-4. DETERMINATION OF SODIUM GLYCOLLATE

### A-4.1 Reagents

**A-4.1.1 Chromotropic Acid Solution** — 5 percent.

**A-4.1.2 Concentrated Sulphuric Acid** — Conforming to IS : 266 - 1961\*.

**A-4.1.3 Standard Sodium Glycollate Solution** — Weigh 0.1000 g of sodium glycollate, dissolve in water and dilute to one litre with water in a measuring flask. One millilitre of this solution contains 0.1 mg of sodium glycollate.

### A-4.2 Procedure

**A-4.2.1 Calibration Graph** — Take 0.1, 0.2, 0.3, 0.5, 0.7, and 1.0 ml of standard glycollate solution in small stoppered conical flasks. Add

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\*Specification for sulphuric acid (*revised*).

sufficient water to each flask to make the volume to 1 ml followed by 0.5 ml of chromotropic acid and 20 ml of concentrated sulphuric acid. Similarly prepare a blank using 1 ml water instead of standard glycollate solution. Then heat all the flasks in a boiling water-bath for 30 minutes, cool to room temperature and make up the volume to 25 ml with concentrated sulphuric acid. Measure the optical densities of the solutions in a spectrophotometer or electrophotometer at a wavelength of 570 m $\mu$  in 1 cm glass cells. Draw a graph plotting optical densities as a function of the quantities of sodium glycollate present in 25 ml of sulphuric acid solution.

**A-4.2.2** Weigh 0.5 g of the dried material ( *see A-5.2.2* ) and dissolve in 100 ml of water contained in a conical flask. Pipette out 1 ml of the solution into 25 ml glass stoppered conical flask. Add 0.5 ml of chromotropic acid solution ( freshly prepared ) and 20 ml of concentrated sulphuric acid. Similarly prepare a blank using 1 ml of water in place of the sample solution. Loosen the stoppers of the flasks and heat in boiling water bath for 30 minutes. Cool to room temperature and make up the volume to 25 ml with concentrated sulphuric acid in a standard flask. Measure the optical density of the solution in the spectrophotometer or electrophotometer at a wavelength of 570 m $\mu$  and by reference to the calibration chart ( *see A-4.2.1* ) read the sodium glycollate content corresponding to this optical density.

#### A-4.3 Calculation

$$\text{Sodium glycollate content,} = \frac{10 m}{M} \\ \text{percent by mass}$$

where

$m$  = mass in mg of sodium glycollate present in 25 ml of sulphuric acid solution, and

$M$  = mass in g of the dried material taken for the test.

### A-5. DETERMINATION OF MOISTURE CONTENT

#### A-5.1 Method A ( Karl Fischer Method )

**A-5.1.1** Weigh 0.5 g of the material and determine the moisture content as described in IS : 2362 - 1973\*.

#### A-5.2 Method B ( Oven Method )

##### A-5.2.1 Apparatus

**A-5.2.1.1 Weighing Bottle** — Squat type, with stopper.

**A-5.2.1.2 Electric Oven** — Maintained at  $105 \pm 2^\circ\text{C}$ .

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\*Determination of water by Karl Fischer method ( *first revision* ).

**A-5.2.2 Procedure** — Weigh accurately about 25 g of the material into a tared, stoppered, weighing bottle. Keep the bottle in an oven maintained at  $105 \pm 2^\circ\text{C}$  for 3 hours. Cool in a desiccator and weigh. *Preserve the dried material for the determination of all other requirements.*

### A-5.2.3 Calculation

$$\text{Moisture, percent by mass} = \frac{100 (M - M_1)}{M}$$

where

$M$  = mass of the sample before drying, and

$M_1$  = mass of the sample after drying.

## A-6. DETERMINATION OF IRON

### A-6.1 Apparatus

**A-6.1.1 Nessler Cylinders** — 50 ml capacity ( see IS : 4161 - 1967\* ).

### A-6.2 Reagents

**A-6.2.1 Hydrochloric Acid** — Conforming to IS : 265 - 1962†.

**A-6.2.2 Ammonium Persulphate** — Solid.

**A-6.2.3 Ammonium Thiocyanate Solution** — Dissolve 300 g of ammonium thiocyanate in water and dilute to 1 000 ml.

**A-6.2.4 Dilute Sulphuric Acid** — 10 percent ( v/v )

**A-6.2.5 Standard Iron Solution** — Dissolve 0.702 g of ferrous ammonium sulphate  $[\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}]$  in 10 ml of 10 percent sulphuric acid and dilute to 100 ml with water. Take 10 ml of this solution, add 10 ml of dilute sulphuric acid and then dilute to 1 000 ml with water. One millilitre of this diluted solution contains 0.01 mg of iron ( as Fe ).

**A-6.3 Procedure** — Weigh accurately 1 g of the material into a Nessler cylinder and dissolve in about 20 ml of water, add 2 ml of hydrochloric acid and dilute up to the mark. Add 30 to 50 mg of ammonium persulphate crystals, 3 ml of ammonium thiocyanate solution and mix. In another Nessler cylinder carry out a control test with 4 ml of standard iron solution and same quantity of other reagents. Compare the colour produced in both the cylinders in good diffused light against a white background.

**A-6.3.1** The limit prescribed in Table 1 shall be taken not to have been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

\*Specification for Nessler cylinders.

†Specification for hydraulic acid ( revised ).

## APPENDIX B

( Clause 5.1 )

### SAMPLING OF SODIUM MONOCHLOROACETATE

#### B-1. GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, storing and handling test samples, the following precautions and directions in addition to those given in 3, shall be observed.

**B-1.1** Samples shall be taken at a place protected from damp air, dust and soot.

**B-1.2** Sampling instrument shall be clean and dry.

**B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.5** The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

**B-1.8** Samples shall be stored in a cool and dry place.

#### B-2. SCALE OF SAMPLING

**B-2.1 Lot** — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

**B-2.2** For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number ( $n$ ) of containers to be selected for drawing the samples shall depend upon the size ( $N$ ) of the lot and shall be in accordance with Table 2.



**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED  
FOR SAMPLING**

*(Clause B-2.2)*

Lot Size <i>N</i>	Sample Size <i>n</i>
(1)	(2)
4 to 25	3
26 „ 50	4
51 „ 100	5
101 „ 150	6
151 „ 300	7
301 and above	8

NOTE— When the size of the lot is three or less, all the containers shall be sampled.

**B-2.3** These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3, ..., up to  $r$  and so on, where  $r$  is the integral part of  $N/n$ ;  $N$  being the number of the containers in the lot and  $n$  the number of containers to be selected. Every  $r$ th container thus counted shall be withdrawn to give sample for test.

### **B-3. TEST SAMPLES AND REFEREE SAMPLE**

**B-3.1** From each of the containers selected as in **B-2.2**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each container shall be approximately equal to thrice the quantity required for testing purposes.

**B-3.2** Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

**B-3.3** From the samples ( **B-3.2** ) representing different containers selected as in **B-2.2** a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, not less than 600 g. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

**B-3.4** The remaining portions of the material in the samples ( *see B-3.2* ) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers selected shall be for the purchaser, another for the supplier and the third for the referee.

**B-3.5** All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **B-1.7**.

**B-3.6** The referee samples consisting of a composite sample and a set of  $n$  individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

#### **B-4. NUMBER OF TESTS**

**B-4.1** Tests for description ( **2.1** ), sodium monochloroacetate content and free monochloroacetic acid content shall be conducted on each of the individual samples.

**B-4.2** Tests for all other requirements given in Table 1 shall be conducted on the composite sample ( *see B-3.3* ).

#### **B-5. CRITERIA FOR CONFORMITY**

**B-5.1 For Individual Samples** — The lot shall be declared as conforming to the requirements of description, sodium monochloroacetate content and free monochloroacetic acid content if each of the test results satisfies the corresponding requirements given in Table 1.

**B-5.2 For Composite Samples** — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample ( **B-4.2** ) the test results for the characteristics shall satisfy the relevant requirements given in Table 1.

# INDIAN STANDARDS

ON

## ORGANIC CHEMICALS ( MISCELLANEOUS ) MATERIALS

IS:

245-1970	Trichloroethylene, technical ( <i>second revision</i> )
501-1963	Oxalic acid, technical and analytical reagent ( <i>revised</i> )
716-1970	Pentachlorophenol ( <i>first revision</i> )
717-1969	Carbon disulphide, technical ( <i>first revision</i> )
718-1970	Carbon tetrachloride ( <i>first revision</i> )
869-1969	Ethylene dichloride ( <i>first revision</i> )
880-1956	Tartaric acid
3321-1973	Formaldehyde solution ( <i>first revision</i> )
4105-1967	Styrene ( vinyl benzene )
4306-1973	Hexamethylenetetramine ( hexamine ) ( <i>second revision</i> )
4566-1968	Methylene chloride ( dichloromethane ) technical
5149-1969	Maleic anhydride, technical
5158-1969	Phthalic anhydride, technical
5254-1969	Acetanilide
5271-1969	Paraformaldehyde
5295-1969	Ethylene glycol
5296-1969	Chloroform, technical and analytical
5297-1969	Perchloroethylene ( tetrachloroethylene ), technical
5341-1969	Benzyl chloride, technical
5464-1970	Citric acid, monohydrate
5573-1969	Ethylene oxide
5591-1969	Chlorobenzene
5592-1969	Monochloroacetic acid
5992-1969	<i>p</i> -Dichlorobenzene, technical
6393-1971	$\alpha$ -Phenylacetamide
6412-1971	Benzoyl chloride, technical
6515-1972	Sodium pentachlorophenate, technical
6712-1972	<i>o</i> -Dichlorobenzene
6716-1972	Benzoic acid, technical
6718-1972	Phenoxyacetic acid
6768-1973	<i>m</i> -Aminophenol
6775-1973	Ethyl chloride, technical
6971-1973	2-ethyl hexan-1-ol
6972-1973	Benzotrichloride, technical
7134-1974	Diphenyl
7135-1973	Dimethyl sulphate, technical
7220-1974	Ethylenediaminetetra-acetic acid, pure and technical
7330-1974	Methods of test for ion-exchange resins

### INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephone : 27 01 31 ( 20 lines )

Telegrams : Manaksanstha

#### Regional offices:

		Telephone
Western :	Novelty Chambers, Grant Road	BOMBAY 400007 37 97 29
Eastern :	5 Chowringhee Approach	CALCUTTA 700072 23-08 02
Southern :	54 General Patters Road	MADRAS 600002 8 37 81

#### Branch offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMEDABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	2 76 49
Kothi No. 90, Sector 18A	CHANDIGARH 160018	2 83 20
5-8-56/57 Nampally Station Road	HYDERABAD 500001	4 57 11
117/418B Sarvodaya Nagar	KANPUR 208005	82 72
B.C.I. Bldg (Third Floor), Gandhi Maidan East	PATNA 800004	2 56 55